

## **General Disclaimer**

### **One or more of the Following Statements may affect this Document**

- This document has been reproduced from the best copy furnished by the organizational source. It is being released in the interest of making available as much information as possible.
- This document may contain data, which exceeds the sheet parameters. It was furnished in this condition by the organizational source and is the best copy available.
- This document may contain tone-on-tone or color graphs, charts and/or pictures, which have been reproduced in black and white.
- This document is paginated as submitted by the original source.
- Portions of this document are not fully legible due to the historical nature of some of the material. However, it is the best reproduction available from the original submission.

DRL NO. 136  
DRD NO. SE-5

9950-658  
DOE/JPL 955733 -81/4  
DISTRIBUTION CATEGORY UC-63

NASA-CR-169062) DEVELOPMENT OF ADVANCED  
CZOCHEKALSKI GROWTH PROCESS TO PRODUCE LOW  
COST 150 KG SILICON INGOTS FROM A SINGLE  
CRUCIBLE FOR TECHNOLOGY READINESS Quarterly  
Progress Report, 1 (Kayex Corp., Rochester, 63/44 N82-26804  
Unclas  
28156

FOURTH QUARTERLY PROGRESS REPORT  
JULY 1 - SEPTEMBER 30, 1981

DEVELOPMENT OF ADVANCED CZOCHEKALSKI  
GROWTH PROCESS TO PRODUCE LOW COST  
150 KG SILICON INGOTS FROM A SINGLE CRUCIBLE  
FOR TECHNOLOGY READINESS

PROGRAM MANAGER: R. L. LANE

KAYEX CORPORATION  
1000 MILLSTEAD WAY  
ROCHESTER, NEW YORK 14624



"The JPL Flat Plate Solar Array (FSA) Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, by agreement between NASA and DOE."

DRL NO. 136  
DRD NO. SE-5

DOE/JPL 955733  
DISTRIBUTION CATEGORY UC-63

FOURTH QUARTERLY PROGRESS REPORT  
JULY 1 - SEPTEMBER 30, 1981

DEVELOPMENT OF ADVANCED CZOCHRALSKI  
GROWTH PROCESS TO PRODUCE LOW COST  
150 KG SILICON INGOTS FROM A SINGLE CRUCIBLE  
FOR TECHNOLOGY READINESS

PROGRAM MANAGER: R. L. LANE

KAYEX CORPORATION  
1000 MILLSTEAD WAY  
ROCHESTER, NEW YORK 14624

"The JPL Flat Plate Solar Array (FSA) Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, by agreement between NASA and DOE."

"This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights."

## CONTENTS

	<u>PAGE</u>
I INTRODUCTION	1
II SUMMARY	3
III PROGRESS	4
A. CONSTRUCTION AND TEST	4
B. PROCESS DEVELOPMENT	4
C. CONTROLS AND AUTOMATION	5
D. ANALYTICAL STUDY	6
E. DOCUMENTATION - ECONOMIC ANALYSIS	8
IV PROGRAM PLAN	12
V COST AND DIRECT LABOR DATA	12
FIGURES	
FIGURE 1 PROGRAM PLAN	13
FIGURE 2 TOTAL INCURRED COST	14
FIGURE 3 DIRECT LABOR HOURS	15
TABLES	
TABLE 1 ECONOMIC ANALYSIS	9

## I INTRODUCTION

This program for "Advanced Czochralski Growth Process to Produce Low-Cost 150 kg Silicon Ingots from a Single Crucible for Technology Readiness" has several goals:

- A. Provide a modified CG2000 crystal grower capable of pulling a minimum of five crystals, each of approximately 30 kg in weight, 150 mm diameter from a single crucible with periodic melt-replenishment.
- B. Crystals to have: resistivity of 1 to 3 ohm-cm, p-type; dislocation density below  $10^4$  per  $\text{cm}^2$ ; orientation (100); after growth yield of greater than 90%.
- C. Growth throughput of greater than 2.5 kg per hour of machine operation using a radiation shield.
- D. Prototype equipment suitable for use as a production facility.
- E. The overall cost goal is \$.70 (1980 \$) per peak watt by 1986.

To accomplish these goals, the modified CG2000 grower and development program includes:

- A. Increased automation with a microprocessor based control system which reduces operator attention and avoids operator errors.
- B. Sensors development which, during the program, will increase the capability of the automatic controls system.
- C. Process development which will: define the process control variables for accelerated growth rate using a radiation shield; analyze variations in the effects of silicon feed material and meltback rate of greater than 25 kg per hour; analyze the effects of these changes on the economic model; investigate and evaluate the effects of process variations on the "quality" of silicon produced by performing purity analysis of the silicon, solar cell fabrication/analysis, and furnace atmosphere analysis.
- D. Provide technology transfer of the developed systems.

To accomplish the above goals, the program has been divided into five general categories:

- A. Construction and Test - to provide a modified CG2000 grower for process development and sensor/automated controls integration.
- B. Process Development - for accelerated growth, accelerated recharge and yield/cost improvement.
- C. Controls and Automation - for sensor development and microprocessor control integration to the Mod CG2000.
- D. Analytical Study - for purity analyses and solar cell fabrication.
- E. Documentation - for reporting, economic analysis, and process specification.

## II SUMMARY

Process development under the extended program was limited to one crystal growth run. The slight twisting of the crystal (corkscrewing) experience during previous runs continued and will require further investigation.

Sensor development resulted in a revised dip temperature sensor viewing angle to minimize spurious reflections from the melt.

The Kayex-Hamco Automatic Grower Logic computer system was integrated to the Mod CG2000 and two test crystals grown. These runs successfully demonstrated the automatic control functions of the AGL system.

The analytical task included trial measurements with the gas chromatograph portion of the Furnace Atmosphere Analysis system. The water and oxygen analyzers will be received next quarter.

Also, automated sampling and curve integration will be added to the gas chromatograph for automated and more accurate readings.

An economic analysis based on present Mod CG2000 process parameters is presented. The present throughput is 1.78 kg per hour. A comparison to 2.5 kg per hour cost shows that a 22% reduction in cost results when the throughput is increased.

The revised program plan is on schedule.



### III PROGRESS

#### A. Construction and Test

The Mod CG2000 was completed in March, 1981. When necessary, modifications to the equipment are reported in other sections.

#### B. Process Development

The revised program plan resulted in a reduced effort on process development during this quarter. The Mod CG2000 grower was fitted with the Kayex-Hameco Automatic Grower Logic computer-based control system and this work is reported in Section III-C.

During the previous quarter (April-June), the Mod CG2000 had several process runs performed for debug of the grower. An Ircon with Kayex electronics diameter controller was installed. Also, a 150 kg run was performed, but was limited in pull speed by a slight twisting of the crystals, a phenomenon usually referred to as "corkscrewing".

During this quarter (July-September), crystal run #12 was made to assess whether the crystal corkscrewing problem was due to a mechanical misalignment. A routine alignment check had indicated that the seed lift cable was not correctly aligned to the crucible shaft. This problem was rectified and the run carried out.

The grower was loaded with 35 kg of recycle silicon in a 15" crucible. Meltdown was achieved in 2-1/2 hours at 94 KW. From the first dip, the crystal produced was zero-D, diameter was 6 inches, and approximately 7" of body growth was made. This growth was interrupted by a plant water pressure drop which temporarily shut down the grower. The crystal was pulled clear of the melt and it was evident that corkscrewing was still present.

The 7" length of crystal was re-dipped with the intention of pulling out as much remaining silicon as possible. Unfortunately, the run was abruptly terminated at an ingot weight of 21.8 kg when the neck broke and the crystal

dropped back into the melt.

Alignment was checked after the run and found to be unchanged. This indicates that the corkscrew problem is pull rate/temperature profile related and therefore fundamental to the particular hot zone configuration being used.

The Mod CG2000 was then prepared for using the Automatic Grower Logic computer system.

Plans are to continue efforts for increasing the growth rate and throughput, using the Automatic Grower Logic (AGL) system, and to conduct more investigative work on the corkscrew problem.

## C. Controls and Automation

### 1. Sensor Development

The test and development program continued with the growth of three four-inch diameter crystals from 18 kg charges in the CG2000 RC development grower. Previously reported efforts had concentrated on 12 kg charges. The use of increased charge size was motivated by the need to check sensor reproducibility with larger charges prior to transferring the sensor systems to the JPL modified 2000 crystal grower.

These tests led to the conclusion that melt temperature sensor reproducibility was less than adequate with the increased charge size. It is believed that the reproducibility was being affected by spurious reflections of hotter surfaces off the surface of the melt. This problem was solved by replacing the two-color pyrometer with a single-color unit, which is less susceptible to errors due to reflections. In addition, the pyrometer geometry was arranged so that it viewed the melt surface at a 90° angle. This minimizes spurious reflections from hotter surfaces.

### 2. Mod 2000 Controls

The Kayex-Hamco Automatic Grower Logic computer-based control system

was connected to the Mod 2000 grower. The system has been tested and verified and two crystals were grown to verify the functioning of the control systems. These runs were conducted prior to completion of the new pyrometer arrangement referred to above.

Growth Run #13: This run was conducted on August 16, 1981. Thirty kilograms of recycled silicon were used. For economy reasons, a 14-inch crucible was used in a 15-inch hot zone. The primary purpose of the run was to verify the function of the shoulder and diameter sensors and to align these sensors under growth conditions. The AGL control system was used to grow two ingot necks and crowns and approximately two inches of 6-inch diameter body under Automatic Diameter Control. The remaining silicon was pulled from the crucible under manual control to expedite removal of the maximum amount of silicon from the crucible prior to grower shutdown.

Growth Run #14: This run was conducted on August 20, 1981 with meltdown, neck, crown and body growth controlled by the AGL computer systems. Melt temperature adjustment to the proper temperature for seed dip was performed manually, as this function was not yet fully implemented in the automatic system. The run was conducted with 30 kilograms of recycled silicon in a 15-inch crucible. 24.5 kilograms were pulled. The run successfully demonstrated the principal automatic control functions of the AGL system.

#### D. Analytical Study

As part of the goals of this task, a gas chromatograph, oxygen analyzer, and hygrometer with an automatic sampling system will be used to monitor the oxygen, water, carbon monoxide and other possible gases.

During this quarter, the problem of varying peak heights with sample hold time was corrected on the gas chromatograph. A leak was detected at a fitting on the coupling valve and was corrected. As expected, the peak

height is now independent of the sample hold time.

Preliminary findings of CO concentration during trial measurements of CG2000 bakeout and growth runs are:

1. Bakeout of graphite parts result in approximately 100 ppm CO. Also, a sharp peak at 17 seconds retention time indicates a light gas, possibly hydrogen, is present.
2. During a growth run, the concentration of CO increases as temperature level increases during meltdown of the silicon. Also, the CO concentration decreases as the temperature is reduced. The 17 second, light gas peak is also observed. Calculated concentrations ranged from 1200 ppm to 5000 ppm.
3. As the crystal grows and the crucible is raised in the hot zone, the CO concentration decreases to approximately 650 ppm - 250 ppm range at the end of the growth cycle.
4. Typically, the CO concentration decreases rapidly when power is turned off.

On August 20, 1981, measurements were made during crystal run No. 14 with the gas chromatograph (GC). Calculated concentrations of CO, based on observed peak heights, matched previous observations. The CO concentrations observed were:

1. At 3.3 kg of crystal - 1450 ppm
2. At 20-23 kg of crystal (near the end of the run) - 650-610 ppm.

These values fall within the range of 650-250 observed near the end of previous runs. It is planned that the Oxygen Analyzer, Hygrometer, and Integrator for the G.C. and Event Control Module will be released from hold and delivered in October and November. The system can then be made more accurate for G.C. measurements by using the Integrator. Also, water and

oxygen measurements, as well as automatic sampling, will be incorporated into the overall system. Calibrated hydrogen gas will be obtained to verify that the light gas peaks observed are actually hydrogen gas.

#### E. Documentation - Economic Analysis

In the previous quarterly report (April-June), economic analyses were presented using the desired throughput rate of 2.5 kg per hour at 6" diameter for 150 kg total pulled weight. The resulting three cases were:

<u>Case</u>		<u>CZ Add-On</u>	
		<u>\$/kg</u>	<u>\$/Peak Watt</u>
1	Pulling 5 crystals, each 30 kg	21.62	0.1525
2	Pulling 4 crystals, each 37.5 kg	21.13	0.1490
3	Pulling 3 crystals, each 50 kg	20.66	0.1457

In this quarter, current Mod CG2000 process parameter results were used to calculate the expected CZ add-on cost:

	<u>CZ Add-On</u>	
	<u>\$/kg</u>	<u>\$/Peak Watt</u>
Pulling 5 crystals, each 30 kg	27.56	0.1944

Table 1 shows the total analysis.

A 22% reduction in cost will result from increasing throughput from 1.78 kg/hr to 2.5 kg/hr.

TABLE 1  
ECONOMIC ANALYSIS

CZ ADD-ON COST BASED ON CURRENT MOD CG2000 PROCESS PARAMETERS

CONDITIONS

Crucible Diameter (in)	15
Crystal Diameter (in)	6
Total Poly Needed (kg)	158
Total Crystal Pulled (kg)	150
Avg. Straight Growth Rate (in/hr)	2.5
Pulled Yield (%)	94.9
Yield After CG (% of Melt)	83.5
Individual Crystal Wt. (kg)	30
No. Crystals/Crucible	5
Cycle Time (hr)	84

PROCESS CYCLE TIMES

OPERATION	TIME (MNS)
-----------	------------

1. PREPARATION

Load Polysilicon	20
Close Furnace	10
Pump Down	20
Meltdown	150
Subtotal	<u>200</u>

2. GROWTH CYCLE (INITIAL)

Lower Seed	*
Stabilize Temp.	40
Neck Growth	20
Crown Growth	70
Straight Growth	562
Taper End	60
Subtotal	<u>752</u>

3. RECHARGE/GROWTH CYCLE (x 4)

Cool Crystal	30
Remove Crystal	10
Load Hopper, Vac. Down (x 2)	60
Lower Hopper (x 2)	10
Dump & Melt	125
Lower Seed	*
Stabilize Temp.	40
Neck Growth	20
Crown Growth	70
Straight Growth	562
Taper End	60
Subtotal	<u>987</u>
(x 4)	<u>3948</u>

TABLE 1 (Cont'd)

4. SHUT DOWN CYCLE

Cool Furnace	80
Remove Crystal	**
Clean, Set Up	60
Subtotal	<u>140</u>

Total Cycle Time (mins) 5040 = 84 hours.

\*Completed during Melt Stabilization Time.

\*\*Completed during Furnace Cooling Time.

GROWTH RATE CALCULATION

Grow Diameter (in)	6.2
Straight Crystal Wt. (kg)	27
Straight Growth Time (hr)	9.37
Avg. Growth Rate (kg/hr)	2.88
Wt. per Unit Length (kg/in)	1.153
Avg. Pull Rate (in/hr)	2.5

SAMICS/IPEG INPUT DATA AND COST CALCULATIONINPUT DATA (\$1980)

1. Capital Equipment Cost [EQPT]	\$ <u>247,560</u>
2. Floor Space [SQFT]	<u>120</u>
3. Annual Direct Salaries	
Prod. Operator (0.65 man @ \$13,160/yr)	8,554
Elec. Tech. (0.3 man @ \$16,940/yr)	5,082
Inspector (0.1 man @ \$11,550/yr)	<u>1,155</u>
Total [DLAB]	\$ <u>14,791</u>
4. Direct Materials Usage Based on Machine Utilization of 85% = 88.6 cycles/yr.	
Crucibles 15" x 12" @ \$300 ea	26,580
Seeds (\$20/cycle)	1,772
Dopant (\$25/cycle)	2,215
Argon (60 ft <sup>3</sup> /hr at \$0.05/ft <sup>3</sup> )	22,320
Graphite (3 sets 15" parts/yr)	<u>26,661</u>
Materials Total [MATS]	\$ <u>79,548</u>

TABLE 1 (Cont'd)

## 5. Utilities

Electricity @ \$0.04/kw hr	
Meltdown @ 100 kw	\$ 3,839
Avg Grow @ 75 kw	18,606
Water @ $\phi 0.7/\text{ft}^3$	
30 gpm for 97% cycle	<u>12,110</u>
Utilities Total [UTIL]	\$ 34,555

IPEG PRICE

5 x 30 kg Crystals

C1 EQPT x \$0.57/yr = \$EQPT	141,109
C2 SQFT x \$109/yr = \$SQFT	13,080
C3 DLAB x \$2.1/yr = \$DLAB	31,061
C4 MATS x \$1.2/yr = \$MATS	95,458
C5 UTIL x \$1.2/yr = \$UTIL	<u>41,466</u>
Total Annual Cost	\$ 322,174
Quan (Total Charged x Yield after CG) kg =	11,689
Add-On Cost \$/kg =	<u>27.56</u>
Add-On Cost $\phi$ /Peak Watt	<u>19.44</u> $\phi/W_p$
(Assumes 1 kg = 1 M <sup>2</sup> )	
Throughput kg/hr =	1.78



#### IV PROGRAM PLAN

The revised program plan is updated and shown in Figure 1.

#### V COST AND DIRECT LABOR DATA

The total incurred cost and direct labor graphs have been updated and are shown in Figures 2 and 3, respectively.

	<u>Previous Total</u>	<u>Current Month</u>	<u>Total To Date</u>
Costs	\$ 541,780	\$ 7,423	\$ 549,203
Man-hours	5,299.1	143.5	5,442.6

ORIGINAL PAGE IS  
OF POOR QUALITY

ORIGINAL PAGE IS  
OF POOR QUALITY

Advanced Czochralski Growth  
For Technology Readiness

Program Plan, Revision No. 2

DOE/JPL 955733

Kayex Corporation  
April 21, 1981

1980 → ← 1981

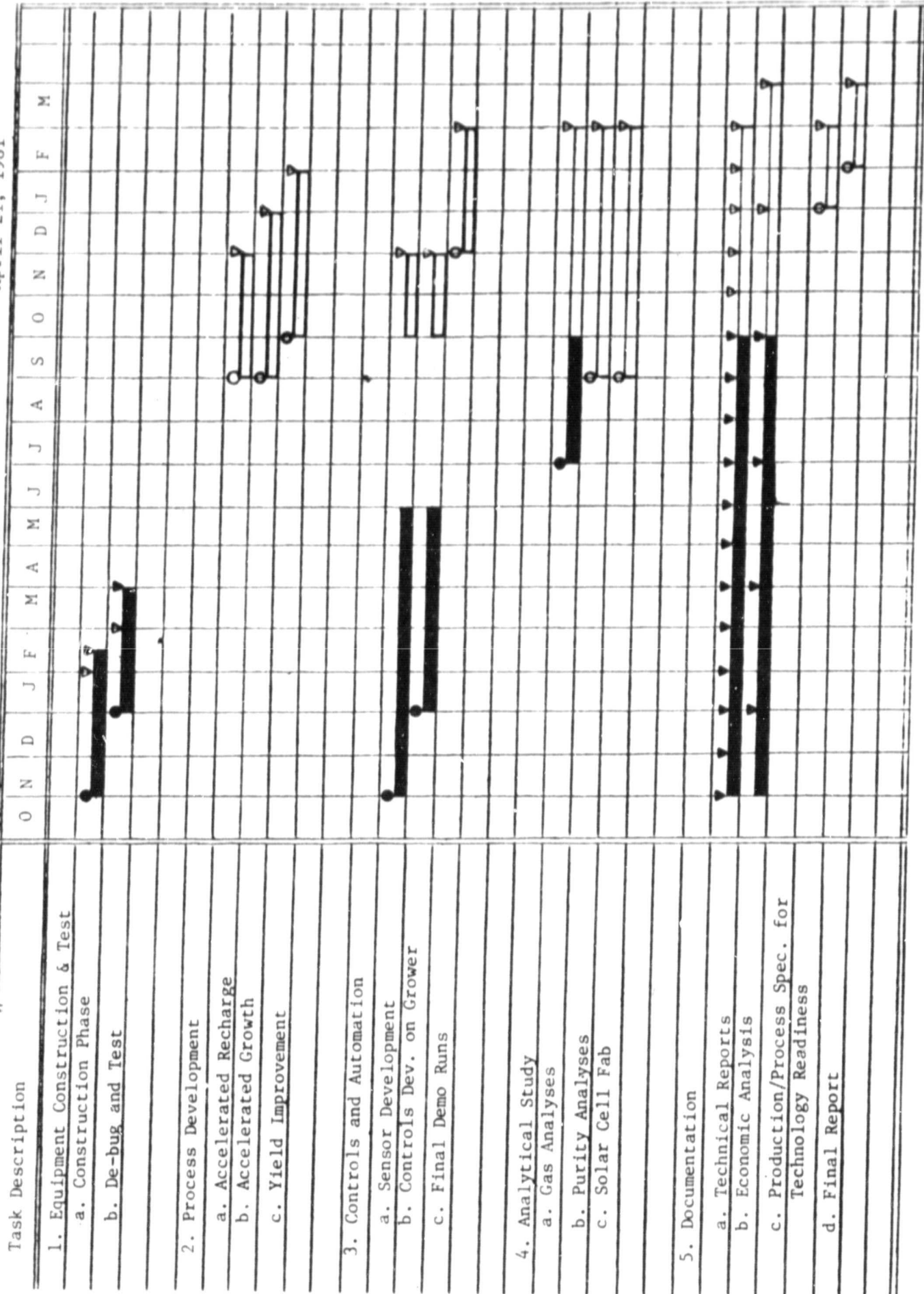
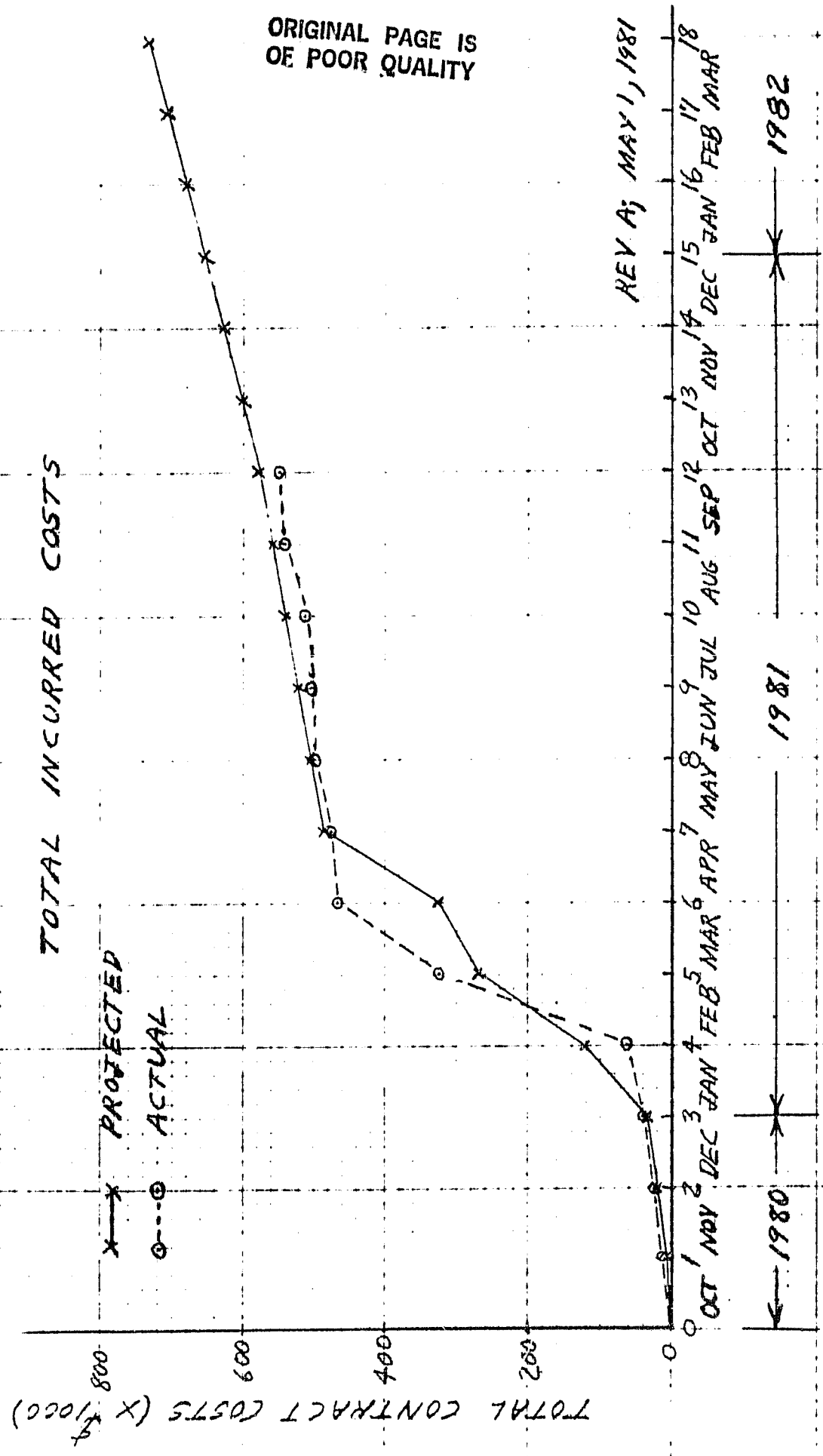


Figure 1

PERN, NIM, VILGAM, J. J. ELIZABETH

ADVANCED C&G GROWTH  
TECHNOLOGY READINESS

KAYEX CORPORATION  
DOE/EPL 955733



ORIGINAL PAGE IS  
OF POOR QUALITY

REV A; MAY 1, 1981

Figure 2  
14

ORIGINAL PAGE IS  
OF POOR QUALITY

KAYEX CORPORATION

DOE/JPL 955733

ADVANCED CE GROWTH  
TECHNOLOGY READINESS

DIRECT LABOR HOURS

PROTECTED  
ACTUAL

DIRECT LABOR HOURS (X 1000)

REVA; MAY 1, 1981

OCT NOV DEC JAN FEB MAR APR MAY JUN JUL AUG SEP OCT NOV DEC JAN FEB MAR  
1980 1981 1982

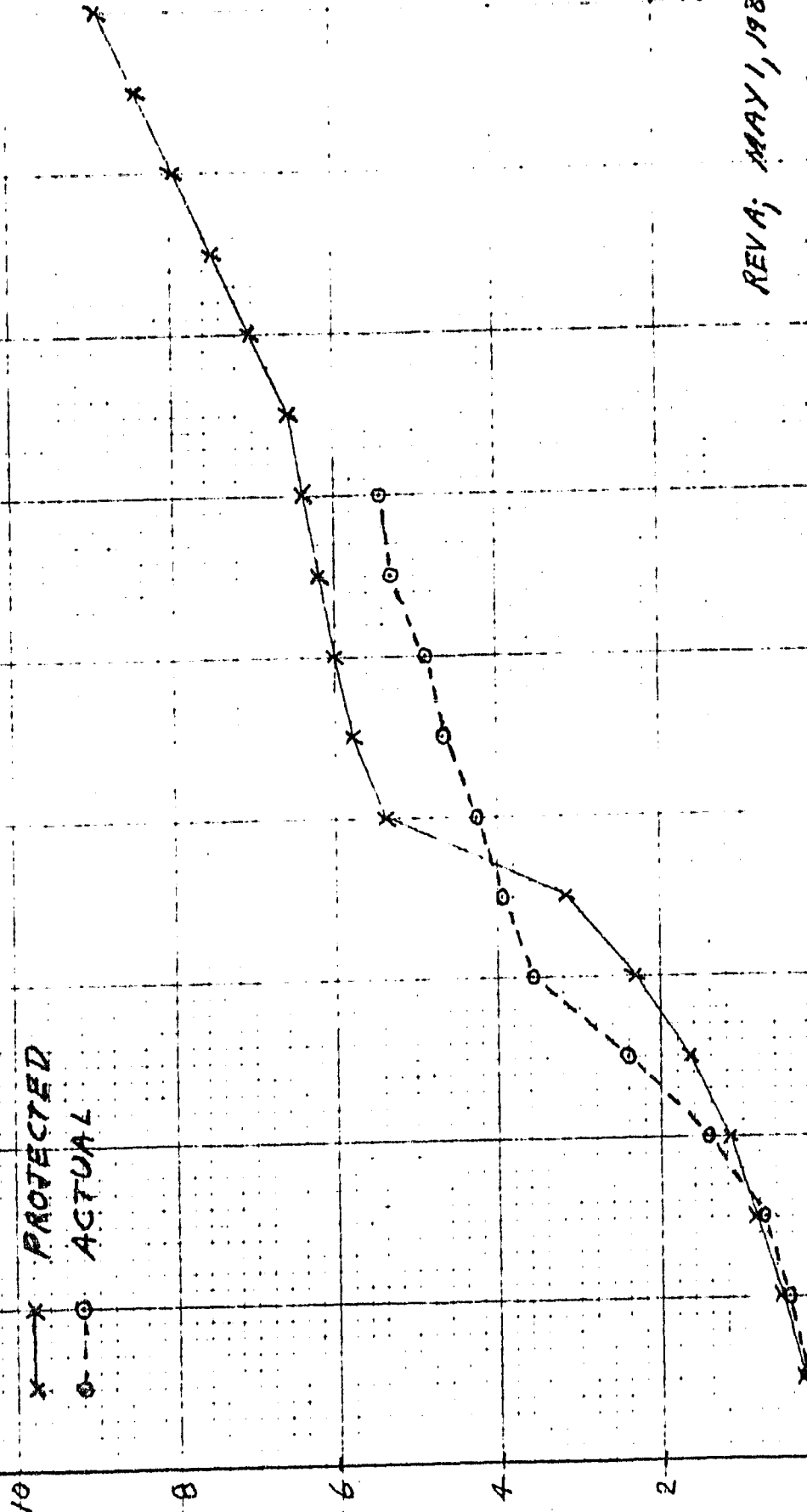


Figure 3